# QUALITY ASSURANCE MANUAL

**Revision 09/2004**

Effective November 1, 2004

Quality Assurance Guidelines Applicable
to all Chemical and Microbiological Testing

**ASSOCIATED LABORATORIES**
**806 N. BATAVIA**
**ORANGE, CA 92868**
**714-771-6900**

**SIGNATURES AND APPROVALS:**

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Robert Webber — Date: 11/8/04

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**DIRECTOR OF QUALITY ASSURANCE:**

James McCall, PhD — Date: 11/08/04

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## MANAGEMENT QUALITY POLICY STATEMENT

It is the policy of Associated Laboratories to provide all clients with test results that are accurate and legally defensible. Associated Laboratories management is committed to good professional practices and quality in environmental testing and calibration as documented in the Quality Assurance Manual and all applicable NELAC standards.

This policy has the full support of Management and must be accomplished with the cooperation of all employees. All personnel concerned with environmental testing and calibration activities within the laboratory are required to familiarize themselves with the quality documentation and implement the policies and procedures in their work.

## ORGANIZATION AND MANAGEMENT STRUCTURE

Associated Laboratories is a privately owned, independent laboratory incorporated in California (DePar, Inc.). The laboratory is actively managed by three directors. The laboratory is organized into Departments as follows:

1. Sample Receiving
2. Sample Custodian and Sample Storage
3. General Chemistry
4. Metals (ICP/AA)
5. Pesticides Analysis
6. Hydrocarbons Analysis
7. Volatile Organic Compounds GCMS
8. Semi-Volatile Organic Compounds GCMS
9. Microbiology
10. Fish Bioassay
11. TOC / Radioactivity
12. Sampling and Sample Pickup
13. QA Department

Each Department is managed by a Department Supervisor who reports to the Laboratory Directors.

The Quality Assurance Department operates independently from the other Departments. The Quality Assurance Director reports directly to the Laboratory Directors.

The Directors manage all operations of the laboratory and are the official signatories for all Laboratory Analysis Reports and other official documents of the Laboratory. The QA Director is the official signatory for Quality Assurance documents and may also sign Laboratory Analysis Reports. The signature page of this document includes all approved laboratory signatories.

## JOB DESCRIPTIONS OF KEY STAFF

The job descriptions of key staff are attached in Appendix A

## FACILITIES AND EQUIPMENT

ASSOCIATED LABORATORIES is located in two buildings:

Main Office and Laboratory: 806 North Batavia Street, Orange, CA 92868

Annex: 1108 West Barkley, Orange, CA.

Telephone: 714-771-6900
Fax No: 714-538-1209

Associated Laboratories has been in operation for over 80 years and is currently employing 75+ personnel.

Our main facility occupies 10,000 square feet, 8,000 square feet is laboratory space and 2,000 square feet office space. The Annex occupies 7,500 square feet and is maintained free of organic solvent vapors for analysis of volatile organic compounds. The annex also contains the microbiology and metals laboratories.

The latest equipment inventory is attached (Appendix D)

## STATE ACCREDITATION

Associated Laboratories is accreditated by the following agencies:

- State of California, Department of Health Services, Environmental Laboratory Accreditation Program, Berkeley, Certificate No. 1338
- State of Hawaii, Department of Health, Safe Drinking Water Branch.
- State of Nevada, Department of Human Resources, Health Division, Bureau of Licensure and Certification.
- U.S. Army Corps of Engineers, Dept. of the Army, Omaha, NE.
- U.S. Food and Drug Administration, Department of Health and Human Services.

Copies of the Certification License and List of Licensed Parameters are available upon request.

## PERSONNEL TRAINING PROGRAM

All current as well as new technical personnel are required to become familiar with the the following documents:

**Laboratory Safety Manual** - A formalized laboratory safety training course has been established, including a video discussion of safety and a written test. An attendance log and the test results are filed in the Employee Safety Documentation File. Each employee is also given a copy of the Laboratory Safety Manual.

**Quality Assurance Manual** - A copy of the Quality Assurance Manual is available in all departments. All employees are required to understand and follow the appropriate Quality Assurance guidelines and procedures.

**Standard Operating Procedures** - Standard Operating Procedures (SOP's) are available to all analysts for most analytical methods. For analytical methods, the SOP provides details regarding specific procedures and QA acceptance limits. SOP's are also available for most laboratory operations. Analysts are required to understand and follow the standard method requirements as detailed in the SOP for each analytical method. Each SOP is reviewed at least annually by the analysts and department manager to insure that the SOP accurately describes the analytical procedure. All SOP's are approved by the department manager and the QA Director.

The Department Supervisor is responsible for ensuring that all department personnel read and understand the Safety Manual, QA Manual, standard methods and appropriate SOP's. Completion of these requirements and all other specific training are documented in the employee training records. Training records are filed in the employee training file maintained for each technical employee. Successful completion of training courses and other formalized training are also filed in the employee training files.

In addition, the following training is conducted:

Technicians are also given on-the-job training for each new method or procedure by the supervisor or an experienced analyst designated by the supervisor. During the training period the supervisor or experienced analyst continues to be responsible for all analytical results produced by the trainee. This training is also documented on the employee's training record.

Competence to perform each analysis is determined by the supervisor's direct evaluation and successful analysis of Lab Control Samples and/or Performance Evaluation Samples.

Periodically, analysts are encouraged to attend outside classes or other relevant training to increase their job knowledge. Attendance at these courses/seminars are also recorded on the training record.

## DEMONSTRATION OF CAPABILITY

For NELAP certified tests a Demonstration of Capability (DOC) must be performed prior to using any test method, and at any time there is a change in instrument type, personnel or test method (NELAC, Quality Systems Revision 16, Appendix C, July 12, 2002).

a) A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared

independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery ($\bar{x}$) in the appropriate reporting units (such as µg/L) and the standard deviations of the population sample ($n-1$) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory must assess performance against established and documented criteria.

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter. In this case locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

f) A certification statement is completed to document the completion of each demonstration of capability. A copy of the certification statement is retained in the personnel records of each affected employee.

## ETHICS POLICY AND DATA INTEGRITY TRAINING

To prevent Data Fraud/Inappropriate Practices, all technical personnel are trained in ethical and legal responsibilities. Examples of Data Fraud are identified below:

a) Inappropriate use of manual integrations to meet calibration or method QC criteria would be considered fraud. For example, peak shaving or peak enhancement are considered fraudulent activities if performed to meet QC requirements.

b) Time travel of analyses to meet method holding time requirements.

c) Falsification of results to meet method QA requirements.

d) Reporting of results without analyses to support the results.

e) Selective exclusion of data to meet QC criteria (i.e. initial calibration points dropped without technical or statistical justification)

f) Misrepresentation of laboratory performance by presenting calibration data or QC data within data reports which are not linked to the data set reported

g) Notation of matrix interference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (e.g. MB or LCS)

The potential punishments and penalties for improper, unethical or illegal actions include immediate dismissal, and possible legal court action

All technical personnel are required to sign an Ethics and Data Integrity Agreement Form. These forms are filed in the QA Office

Internal audits are performed periodically which include monitoring of data integrity. Any allegations of improper reporting or manipulation of data are investigated promptly.

## DOCUMENT CONTROL AND RECORD KEEPING

All documents relating to laboratory analyses and reporting are kept a minimum of seven years. After that time the records will be destroyed, unless special arrangements are made.

The laboratory maintains a tracking system for Standard Operating Procedures, MDL determinations, training documentation and corrective actions.

A Lab Request is created by the Laboratory LIMS system for each group of samples received from a client to enable organization and tracking of the analyses and final reporting. All analytical results are reported in the LIMS database system, including date of analysis and analyst initials. All documentation other than bound laboratory notebooks relating to the analyses of a client's samples including a copy of the final report, Chain of Custody, all sample preparation worksheets and analytical raw data is attached to each Lab Request. Lab Requests including all relevant data are filed for a minimum of seven years. Other relevant analysis data may be written in bound laboratory notebooks which are maintained in each laboratory department. All calibration data and other relevant data such as calibration checks, which may apply to multiple Lab Requests are filed and retained in the individual departments.

### Corrections

All generated data is recorded in permanent ink. Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction

## REVIEW OF NEW PROJECTS

New projects and contracts are reviewed by laboratory management to ensure that the laboratory has the technical capability and resources to meet the requirements. Any potential conflict of interest or other problem noted in the review is discussed with the client prior to acceptance of the contract or samples.

## PROTECTION OF CLIENT CONFIDENTIALITY

Associated Laboratories recognizes the importance of client confidentiality. Each Lab Report contains the following statement: "The reports of Associated Laboratories are the confidential property of our clients and may not be reproduced or used for publication in part or in full without our written permission. This is for the mutual protection of the public, our clients, and ourselves."

## SAMPLE RECEIVING AND CUSTODY

All sample receiving and log-in is handled by the Sample Receiving Department.

1. All samples are assigned a laboratory identification number during the log-in process. This number is a unique identifier assigned by the laboratory LIMS system.

2. All samples received from a client on the same day on the same Chain of Custody (COC) are normally grouped together in a unique Laboratory Request Number. The Laboratory Request Number is also assigned by the laboratory LIMS system.

3. A Laboratory Request Summary is prepared which includes: date, client name, client sample ID, corresponding laboratory sample number, all analyses to be performed, laboratory area designations and other special instructions.

Procedures for sample receiving and chain of custody for samples are detailed in the Sample Receiving SOP, attached to this document as Appendix B.

## SAMPLE HANDLING PRACTICES AND CHAIN OF CUSTODY

1. After samples are logged in, they are transferred to the Sample Custodian.

2. All transfer of samples out of and into storage are documented on the Sample Control Record Book.

3. The temperature of each refrigerator used for sample storage is monitored each working day, and recorded on the Temperature Control Record. This record is attached to each refrigerator. When the record is completely filled in, it is filed for future reference. If the temperature is out of control limits, the laboratory manager must be notified immediately.

4. Unless notified in writing, all samples will be discarded by appropriate disposal protocol 30 days from the date reported. Samples are discarded in the designated hazardous waste disposal containers. These containers are picked up periodically by a hazardous waste disposal company.

## SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

In general, the shorter the time that elapses between collection of a sample and the analysis, the more reliable will be the analytical results. Preservation is necessary when the interval between sample collection and analysis is long enough to produce changes in either the concentration or the physical state of the constituent to be measured. Preservation of samples is specified in many EPA methods and when possible is confirmed by the laboratory during the sample log in process. The holding time of an analysis is the maximum time that samples may be held before analysis for the analysis to be considered valid. Each department is familiar with the holding times for sample analysis which they perform. The supervisor is responsible for ensuring that these holding times are met for all analyses. If holding times are not able to be met, then every effort is made to notify the client and if necessary send the samples to another laboratory.

Appendix C contains sample container guidelines and holding times as specified by the USEPA for environmental samples.

## LABORATORY LIMS SYSTEM

### Laboratory Information Management System (LIMS)

The laboratory information management system (LIMS) is a client-server network of computers used to login samples, track samples during and after analysis, and report the final results to the client. In addition the LIMS software which is database driven is able to generate historical reports and trends and generate other types of reports such as electronic deliverables which are increasingly used by clients to transfer data into their own computer systems without having to do manual data entry. The LIMS system is also used to track laboratory data such as detection limits (MDL) and reporting limits for analytes.

The hardware components of the LIMS include two servers and approximately twenty-five PC compatible computers running Windows 98 - 2000. The LIMS Software consists of Varian Starlims 7.0 with an Oracle 7 database system.

Security consists of a password login system and nightly tape backups. All reports are reviewed and signed by designated managers before release to the client. Tracking reports are generated daily from the LIMS system to insure timely analysis and reporting of all client samples.

Electronic Delivery Capabilities - laboratory data can be delivered to the client in electronic data deliverable (EDD) formats such as: spreadsheet (Lotus, Excel); standard database file formats (dB, Paradox, etc); delimited or fixed field formatted ASCII; or word processing formatted. The data files can be transmitted to the client either by diskette or directly using e-mail or FTP protocols.

## STANDARD TEST METHODS

Essentially all laboratory analyses are conducted using published standard methods. Standard method sources which are available for use are listed below.

**Analytical Standard Procedures for Environmental Analyses:**

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79- 020,3/1983

Standard Methods for the Examination of Water and Wastewater (American Public Health Association)

40 CFR, Appendix A to part 136-Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (600-series methods)

Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA-600/R-95/131, August 1995. (500-series methods)

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993

Methods for the Determination of Metals in Environmental Samples, Supplement I EPA/600/R-94/111, May 1994

Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition.

NELAC Quality Systems Revision 16, July 12, 2002.

**Analytical Standard Procedures for Food, Feeds, Oil/Fats and Pharmaceuticals:**

Association of Official Analytical Chemists (AOAC).

The American Oil Chemists' Society (AOCS).

Methods of the U.S. Department of Agriculture (USDA).

FDA Pesticide Analytical Manual (PAM).

US Pharmacopeia/National Formulary (USP/NF).

Food Chemicals Codex (FCC).

American Society for Testing and Materials (ASTM)

## STANDARD OPERATING PROCEDURES

Standard Operating Procedures (SOP) are available for most methods to indicate specific procedures, instrumentation, data needs and laboratory data quality requirements. Standard Operating Procedures are available to the analyst and are updated at least annually to insure that method and quality assurance requirements are being met. The original version of the SOPs are filed in the QA Department and controlled copies made available to the department. An inventory list of all current SOP's is maintained by the QA Department.

Each test method shall include or reference where applicable:

1) identification of the test method;
2) applicable matrix or matrices;
3) detection limit;
4) scope and application, including components to be analyzed;
5) summary of the test method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measures;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and,
23) any tables, diagrams, flowcharts and validation data.

## TRACEABILITY OF MEASUREMENTS

Traceability of measurements is achieved by using standards for calibration and calibration checks which are traceable to primary NIST standards. Certificates of Analysis or purity are kept on file for each standard purchased, showing the traceability of the standard to a primary NIST standard. All balances are calibrated and certified annually using NIST certified weights. Thermometers are also calibrated at least annually using a thermometer certified against an NIST temperature standard.

When standard solutions, spiking solutions and calibration check solutions are prepared, the following information is recorded in a Standards Traceability Notebook maintained by each Laboratory Department:

a. The identifying name of the Working Standard consists of the Working Standard Identification and the date of preparation. This name must be unique and apply to only one standard solution, such that the standard can be unequivocally traced back to the date of preparation, analyst and identification of all original standards and reagents used to prepare the standard.

b. Date and analyst initials

c. The name, manufacturer and lot number of each analytical standard, reagent and acid used in the solution.

d. The volume of each standard, reagent and acids used, and the final volume of the solution.

e The calculated concentration of all analytes in the final solution

The final standard solutions are transferred to a storage container and labeled with the identifying Working Standard ID, date of preparation, expiration date, concentration and initials of the analyst who prepared the solution.

All commercially prepared standards have a maximum expiration date of one year from the date of receipt or other expiration date as established and documented by the supplier.

Reagents are purchased from established commercial suppliers as specified by the laboratory standard methods or SOP. Reagents are stored at the appropriate temperature (refrigeration, freezing, room temp) as specified by the supplier.

Lot numbers of reagents are recorded on sample preparation log sheets or in analysis log books to enable traceability.

## CALIBRATION AND VERIFICATION PROCEDURES

### Initial Calibrations

Criteria for Initial Calibrations are specified in the applicable method and Standard Operating Procedure for each method.

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics are included or referenced in the test method SOP

b) Sufficient raw data records are retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, analyst's initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration

c) Sample results must be quantitated from the initial instrument calibration and may not be

quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method, or program.

d) All initial instrument calibrations must be verified with an **Initial Calibration Verification** standard (ICV) obtained from a second manufacturer or lot number. Traceability is to a national standard, when available

e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference. The criteria used must be appropriate to the calibration technique employed.

f) Results of samples outside of the concentration range established by the initial calibration must be reported with defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the detection limit (mdl).

g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable initial instrument calibration are reported with appropriate data qualifiers.

h) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by the laboratory, unless these concentrations are below the laboratory's demonstrated detection limits

i) The number of points for establishing the initial instrument calibration are determined by the method and regulatory guidelines and are stated in the SOP for each method.

**Continuing Calibration Verification (CCV)**

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch

c) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records must explicitly connect the continuing verification data to the initial instrument calibration.

d) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. If the laboratory has not demonstrated acceptable performance, sample analyses shall not occur until a new initial calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

1) when the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted

2) when the acceptance criteria for the continuing calibration verification are exceeded low, i.e., *low bias, those sample results may be reported if they exceed a maximum regulatory* limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

## METHOD DETECTION LIMITS

Method Detection Limits (MDL) are normally determined by taking seven or more aliquots of a sample containing the compounds of interest at a concentration 1 to 5 times the estimated detection limit and processing each sample through the entire analytical method. The MDL is calculated from the standard deviation of the replicate measurements (MDL = 3.143 x Standard Deviation for seven replicate measurements). MDL studies for each method are normally performed at least annually or when a major modification is made to the method or instrumentation used for analysis. Reference: 40 CFR, Ch. 1, Part 136, Appendix B (7-1-86 Ed.).

Method Detection Limits are updated in the laboratory information management system (LIMS) and tracked by the QC Department. The SOP for determination of MDL is attached (Appendix E).

## ANALYTICAL RESULTS REPORTING

Final Reports issued to clients contain at a minimum the following information:

Client name, address, Client ID number and contact or project manager

Dates of sample receipt at the laboratory, date of sampling and date of analysis
Cross reference of lab identification and client sample identification.

Results of analysis for each sample, including the test method, analyte, result, detection limit, date analyzed and analyst initials.

Original copy of the chain-of-custody.

Signature of laboratory manager.

A statement is included in the final report:
*"The reports of the Associated Laboratories are confidential property of our clients and may not be reproduced or used for publication in part or in full without our written permission. This is for the mutual protection of the public, our clients, and ourselves."*

**DATA REVIEW**

All data generated from each analysis are recorded either in a bound laboratory notebook or on worksheets which are attached to the Lab Request package.

Copies of the lab notebook page(s), worksheets, instrument readouts, chromatograms, QC forms and other data pertinent to the analysis are attached to the Laboratory Request Sheet.

In addition to the analytical results and calculations, the manufacturer and lot number of all reagents used must be included. Also the assigned code numbers of all prepared reagent and standard solutions are included for traceability purposes.

The review process includes at least three separate review stages:

The analyst reviews all data and calculations and also checks data for completeness and that any special requirements have been met.

The Lab Supervisor reviews the results and initials the report to signify his/her approval.

After the final report is completed, the Laboratory Manager or signatory of the report reviews the final report and signs the report to signify his/her final approval.

The QA Department reviews a proportionate amount of all QC data generated (at least ten percent) and also reviews all corrective action reports that are submitted by the Departments.

A copy of the test report and all supporting raw data for each Lab Request are maintained on file by the laboratory.

The minimum period of retention for the records is seven (7) years.

**PROCEDURE FOR HANDLING CUSTOMER'S COMPLAINTS**

Associated Laboratories encourages feedback from customers. Complaints such as improper billing or incorrect sample identifications are normally handled by client project managers, who

make every effort to resolve the problem as quickly as possible. Where the complaint involves problems which can not be readily corrected, then the customer's complaints are recorded on a Customer Complaint Form which contains the following information:

Date of complaint
Name of company
Name of person submitting the complaint
How the complaint was submitted
Name of person receiving complaint by phone
Nature of complaint
Department(s) involved

The customer's complaint form is submitted to the department(s) involved for investigation and resolution of the complaint.

The results of the investigation and resolution of the complaint are recorded on the complaint form, signed and dated by the individual handling the complaint and submitted to the Lab Manager to be reviewed and approved.

The customer is notified of the results of the investigation and resolution of the complaint by the Lab Manager or by a person authorized by the Lab Manager, either verbally, by phone, or in the form of a letter.

The Complaint Form and all other documents pertinent to the complaint are filed in the Complaint File maintained by the QA Department.

## QUALITY ASSURANCE PROCEDURES

Quality Control samples are normally analyzed with each batch of samples for each analysis. For environmental samples the Quality Control samples include a Method Blank (MB), Laboratory Control Sample (LCS) and a Matrix Spike and Matrix Spike Duplicate. These QC samples are included in each batch of twenty samples or less for each matrix (frequency equivalent to 5% of all samples analyzed). If spike analyses are not feasible, a duplicate sample analysis is generally performed (eg TDS, dissolved oxygen, turbidity).

1. The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank is processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures are included in the method to determine if a method blank is contaminated. Any affected samples associated with a contaminated method blank are reprocessed for analysis or the results reported with appropriate data qualifying codes.

2. The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicate that the analytical system is "out of control". Any affected samples associated with an out of control LCS are reprocessed for re-analysis or the results reported with appropriate data qualifying codes. The Laboratory Control Sample (LCS) is run at the same frequency as QC samples for each type of matrix. The LCS is obtained when

possible from a source external to the laboratory. The LCS may be prepared by the laboratory using standards from a different source or a different lot number from the source used for calibration standards.

3. A Matrix Spike and Matrix Spike Duplicate sample are normally analyzed with each batch of twenty samples or less. Matrix spikes are duplicate aliquots of a sample which are spiked with the analytes of interest and taken though the same analytical procedures. The recovery of the analyte concentration is calculated to indicate the accuracy of the analysis in the sample matrix The relative percent difference between the Matrix Spike and Matrix Spike Duplicate sample provides a measure of precision of the analyses in the sample matrix.

4. Surrogate spike analyses are performed for all organic analyses when required by the method. Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix. The surrogate spike solution is added to all samples, standards and blanks. The results are compared to the acceptance criteria as published in the mandated test method or laboratory generated acceptance criteria. Results reported from analyses with surrogate recoveries outside the acceptance criteria should include appropriate data qualifiers.

5. All other QC requirements (tuning, multiple points calibration, daily calibration check, etc.) are performed as specified in the method.

6. All QC data are to be recorded on the appropriate forms and kept on file by each department Copies of these forms must be attached to the Lab Requests for all samples associated with that particular QC sample. Accuracy and precision data may be used to generate control charts.

7. Acceptance limits for QC samples are detailed in the Standard Operating Procedure for each method, and may be established by the original reference source or statistical analysis of the historical data for each type of QC sample, method and matrix using control charts.

8. When QC acceptance criteria are exceeded, corrective actions are to be taken as specified in the method or as instructed by the Department Supervisor.

9. Non-conformances such as QA limit failures which cannot be corrected by re-analyses, client requirements which cannot be met or standard method modifications are documentated by initiating a Non-Conformance Document Form (NCD). Appendix F describes the use of the Non-Conformance Document Form.

**Quality Control Procedures for Microbiology**

A complete record of the preparation of each batch of media is maintained in the Media Preparation Log Book. For each batch of media the following information is recorded: date; type of media; lot number and expiration date of dried media; preparer's name; weight of dry media; volume of solution; pH measurements and adjustments; results of control tests.

A sterilization record of each batch of media is maintained in the Sterilization Log Book. The date, media or item identification, time in/out, maximum temperature, total time, sterilization time, controls and maintenance is recorded. Sterilization indicator tape is applied to each batch of prepared media.

The pH of each batch of media is checked either before or after autoclaving and recorded in the Media Preparation Log Book.

Each batch of media is checked using an incubated positive control, negative control and sterility check sample. If any QC check sample fails, the batch is not used for sample analyses.

A duplicate analysis is run on 5 percent of the samples of each type, or at least one per test run.

Quality Control on multiple tube dilution tests:

For routine analyses, the "Completed Test" is run on 10% of positive samples. If there are no positive results from potable water samples, at least one positive source water is run by the completed test quarterly.

For public water supply samples with a history of heavy growth without gas in Presumptive-Phase tubes, all tubes are submitted to the Confirmed Phase to check for coliform bacteria.

## QUALITY ASSURANCE DEPARTMENT FUNCTIONS

### Auditing Activities

Various types of internal audits are performed on Laboratory activities on a routine basis. These audits should reflect as closely as possible, the Laboratory performance under normal operating conditions.

Performance Audits: Evaluation of data reports generated by the laboratory. All technical, clerical and administrative aspects of the data report are reviewed. Errors observed during these ongoing audits are categorized as they relate to the technical accuracy and legal defensibility of data.

Internal audits of each department are conducted at least annually. Routine quality control checks, for example checking laboratory notebooks, daily calibrations, quality control sample frequency are also done on a random basis. Results of internal audits are documented in the internal audits files and reported to management.

A system audit is the physical inspection and review of the entire laboratory operation to verify compliance with the QA Program objectives as stated in the Laboratory's QA Manual. System audits are conducted periodically by external auditors, such as state regulatory agencies, commercial clients or independent auditors representing these clients or agencies.

In response to deficiencies or recommendations from auditing activities, corrective actions reports are required to document the corrective actions taken to correct the deficiencies.

## External Proficiency Testing

The QA Department is responsible for organizing Proficiency Testing (PT) Programs, including WS and WP Studies, and other studies as required by accrediting agencies.

Proficiency Testing samples are obtained from NELAP approved external sources on a semi-annual basis. Results must be satisfactory (within acceptance limits) or a corrective action report is initiated. Proficiency Testing samples are analyzed semiannually or more often for all NELAP accredited tests. PT samples for ELAP accredited tests may be analyzed annually or semiannually. To demonstrate proficiency under NELAP guidelines, the laboratory must pass two of the three most recent PT samples for each accredited test.

## Corrective Action Reports and Departures from Documented Policies

A Non-Conformance Document (NCD) may be required when certain Quality Control criteria are exceeded in a sample analysis batch.

1. Non-conformances such as a sample exceeding holding time, QA limit failures which can not be corrected by re-analyses, client requirements which cannot be met, or standard method modifications are documented by initiating a Non-Conformance Document Form (NCD). A copy of the NCD Standard Operating Procedure and Form is attached (Appendix F).

2. The NCD form is initiated by the analyst in the event of a sample exceeding holding time, Quality Control sample results outside control limits or other known non-conformance to the analytical method or client requirements. The NCD form may also be initiated by the project manager or department manager in the event client requirements are not met or other analytical problems are discovered.

3. After the NCD Form is initiated, the corrective action, if any, must be agreed upon by the department manager or supervisor and the QA Manager. This is documented and signed by the department manager in the second part of the NCD Form. The form is then forwarded to the QA Manager

4. The QA Manager then completes and signs the final part of the form. If necessary, verification of the corrective action is documented in this section

5. A copy of the form is included in the affected data package or the client is notified as appropriate. The original is filed in the Corrective Actions File which is maintained by the QA Manager

When there are deviations from the requirements by the specific method, such as insufficient sample volume, improper preservation, the client should be notified as soon as possible If the client agrees to the deviation, then an explanation of the deviation or non-compliance is required to be attached to the data package and final report.

**Laboratory Standard Operating Procedures and QA Manual**

The QA Department is responsible for ensuring that all Laboratory Standard Operating Procedures and the QA Manual are current. A tracking system is in place to ensure that copies of Standard Operating Procedures are controlled such that only current approved versions are in use in the laboratory.

## PREVENTIVE MAINTENANCE

Written records are kept for each analytical instrument to document inspections, maintenance, troubleshooting, or modifications. Records contain the date, nature of the problem, repair/corrective action taken and the name of the person performing the work. A Maintenance Log Book may be kept for each individual instrument for the purpose of recording any maintenance, repairs, and other associated downtime.

Operational performance of analytical instrumentation is monitored by daily, documented performance checks and calibration verifications in accordance with the Standard Operating Procedures for each type of instrumentation.

Equipment such as analytical balances, ovens, refrigerators and water baths are checked daily for performance within acceptance limits. This information is recorded in a log book maintained for the equipment. Weights used to check the balances are traceable to NIST standards. In addition all balances are inspected and certified by a licensed specialist at least annually.

## QUALITY ASSURANCE MANUAL REVISION HISTORY

Revision 09/2004: QA Manual all sections re-written to incorporate NELAC guidelines.
Added sections for:
- Demonstration of Capability
- Review of New Projects
- Protection of Client Confidentiality
- Calibration and Verification Procedures

Updated Appendix A, Laboratory Job Descriptions
Updated Appendix B, Standard Operation Procedures for Sample Receiving
Updated Appendix D, Equipment Inventory

**APPENDIX A**

**LABORATORY JOB DESCRIPTIONS**

**Technical Director (Lab Director)**

Education: Bachelors degree or equivalent in the chemical, environmental, biological sciences, physical sciences or engineering, with at least 24 college semester credit hours in chemistry.

Experience: At least two years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A masters or doctoral degree in one of the above disciplines may be substituted for one year of experience

Job Description: The technical director(s) means a full-time member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results. This person's duties shall include, but not be limited to, monitoring standards of performance in quality control and quality assurance; monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data.

Responsibilities: Overall responsibility for management of all laboratory operations.

**Quality Assurance Manager**

Education: Bachelor's degree in chemistry or other scientific/engineering discipline or equivalent experience.

Experience: Three or more years experience in a chemistry laboratory.

Job description: The quality manager (and/or his/her designees) shall:

1. Serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2. Have functions independent from laboratory operations for which they have quality assurance oversight;

3. Be able to evaluate data objectively and perform assessments without outside (e g., managerial) influence;

4. Have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC;

5. Have a general knowledge of the analytical test methods for which data review is

performed;

6. Arrange for or conduct internal audits as per 5.4.13 annually; and,

7. Notify laboratory management of deficiencies in the quality system and monitor corrective action.

Responsibilities: Overall development and management of the laboratory quality assurance system as defined by California Dept of Health / ELAP and NELAP requirements.

**Laboratory Supervisor**

Education: Bachelor's degree in chemistry or other scientific/engineering discipline or equivalent experience.

Experience: Three or more years experience in a chemistry laboratory.

Job Description: Responsible for the overall technical and personnel management of a laboratory area or work group. This includes:

1. Interfacing with and taking direction from the Department Head or immediate supervisor.

2. Proper training of personnel in analytical techniques, reporting, quality, assurance and lab safety.

3. Maintaining the orderly flow of work and the timely analyses of samples.

4. Organizing and assigning work duties of the group supervised.

5. Checking QA/QC records for completeness and proper frequency.

6. Providing for technical expertise as required in the group or department.

7. Evaluating and working to constantly improve the quality of data that is being generated (including QA data)

Responsibility, Supervisors are ultimately responsible for:

1. The accuracy, completeness and integrity of all analyses completed by their group or department.

2. Safe practices of their employees.

3. Maintaining effective communication with their employees and upper management of the laboratory.

4. Complete documentation of all analyses and related QA/QC.

5. Any deviation from standard methods or laboratory standard operating procedures

**Chemist**

Education: Requires minimum of Bachelor's degree in chemistry or equivalent experience

Experience: One or more years experience in a chemistry laboratory

Job Description: Conducts analyses in laboratory using standard methods (EPA,AOAC, USP ASTM ,or in-house methods). Must understand lab nomenclature and be proficient in the use of standard lab equipment such as pipets, balances, separatory funnels, burets, etc. Must be able to understand and develop written procedures and SOP's. Must be understand the importance of good lab practices and quality assurance and be able to evaluate the quality of data that is being generated (including QA data). Should be able to work with little supervision.

Responsibility: Chemists are responsible for the accuracy, completeness and integrity of all work that they have been assigned. A chemist should be able to do some trouble-shooting or method development, but any problems must be communicated to their immediate supervisor All deviations from standard methods must be approved by the lab supervisor.

**Analyst**

Education: Requires minimum of Bachelor's degree in chemistry or any scientific/engineering discipline or equivalent experience.

Experience: Once or more years experience in a chemistry laboratory operating and maintaining analytical instrumentation such as AA, ICP, GC, HPLC, etc.

Job Description: Conducts analyses in laboratory using specialized analytical equipment. Analyses are done using standard protocols such as EPA, EPA/CLP, or in-house SOP's) Must understand the theory, use and maintenance of specialized analytical equipment. Must be able to follow written procedures and SOP's and calculate final results, including QA results. Must understand the importance of good lab practices and quality assurance and be able to evaluate the quality of data that is being generated.

Responsibility: Analysts are responsible for the accuracy, completeness and integrity of all work that they have been assigned. If they have questions or problems, this must be communicated to their immediate supervisor. No deviations from standard methods are permitted unless approved by the lab supervisor.

**Lab Technician**

Education: Requires high school diploma with one year of chemistry course work or one year of Chemistry course work or one year experience in a laboratory.

Experience: One or more years experience in a laboratory ( preferably a chemistry lab)

Job Description: Conducts analyses in laboratory using standard methods ( EPA, AOAC, USP, ASTM, or in-house methods). Must understand lab nomenclature and be proficient in the use of standard lab equipment such as pipets , balances, separatory funnels burets, etc. Must be able to follow written procedures and SOP's and calculate final results. Must understand the importance of good lab practices and quality assurance.

Responsibility: Lab Technicians are responsible for the accuracy, completeness and integrity of all work that they have been assigned. If they have questions or problems, this must be communicated to their immediate supervisor. No deviations from standard methods are permitted unless approved by the lab supervisor.

**APPENDIX B**

**STANDARD OPERATING PROCEDURE FOR SAMPLE RECEIVING**

**TABLE OF CONTENTS**

## I. INITIAL RECEIPT OF SAMPLES

This section describes how samples are received and logged into the laboratory. "Logging" refers to the process of documenting receipt of each sample, verification of the analyses requested and entry of information about the sample into the laboratory computer system (LIMS). The sample logging process generates one label for each sample container, a Lab Request Summary on blue paper and a blue Results Worksheet for each department. A copy of the Lab Request Summary and the blue Results Worksheet is transferred to each department which will be analyzing the sample. No sample is analyzed without being properly logged into the laboratory data system, even if the sample is not to be billed.

### A. Handling of Samples Received by Client Delivery:

When a client delivers a sample for analysis, it is important that information about the sample be as complete as possible. This is best done with a properly completed and signed Chain of Custody form. The following information must be obtained before the sample can be accepted:

1. Client's name and address

2. Person to contact regarding the sample(s) and phone number (also fax number if information is to be faxed).

3. Method of payment, does client have an account? If client does not have an account, payment will have to be in advance or "pickup and pay". If the client has an account, a purchase order number is often needed.

4. If the Client wishes to open an account, the accounting department should be notified to be sure the client receives the proper forms and information, this is currently handled by Bill Utter.

5. Before entering a new client into the computer system a unique account code number must be obtained from the accounting department or office supervisor.

6. Both the client and lab employee receiving the sample must both sign the completed Chain of Custody form. The Chain of Custody will normally contain detailed information on the samples. Refer to Section II for a list of required information to be included on this form.

7. The client receives the pink copy of the Chain of Custody. The other copies are attached to the Lab Request Summary.

### B. Sample pick-up by our personnel:

1. All samples received from our drivers should be accompanied by a completed Chain-of-Custody form - signed by the client and by the driver.

2. All coolers received must have a temperature reading immediately upon opening.

a. This reading will be taken by placing the metal probe of the thermometer either into a temperature blank (if provided) or between the respective samples and the cooling media (ice, dry ice, or blue ice).

b. The thermometer should remain in place for 60 seconds to ensure a proper reading.

c. The exact temperature will then be read from the thermometer. The temperature should be in the range of 2 - 6 degrees C.

d. The temperature will be noted on the Cooler Receipt Form.

3. The Chain of Custody and samples must be checked to make sure that all information is in agreement.

4. When the driver relinquishes the samples to the Sample Receiving Department, he or she must require that the Associated Laboratories Chain of custody be signed by an employee of the Sample Receiving Department. A cooler receipt form must be filled out for all coolers received by the Department.

5. All samples brought to the laboratory by a driver will remain under his or her custody until the Associated Laboratories Chain of custody is signed by an employee of the Sample Receiving Department.

6. The pH of all chemically preserved aqueous samples, except volatile samples, must be checked and documented upon receipt at the laboratory. If discrepancies are noted, the laboratory must contact the project manager or client immediately. The pH is measured and reported on a pH reporting form. This form is attached to the Chain of Custody.

7 Any problems with improper preservation, sample container type, volumes, etc. are to be noted on the Cooler Receipt Form. This is to document problems which may interfere with a proper analysis of the sample. The project manager should be notified so that the client can be contacted as soon as possible.

8. Information on the sample pickup is also logged into the bound Driver's Logbook.

9. All organic volatile samples (VOA) must be stored in the Sample Receiving refrigerator until they are labeled.

10. All information is checked to be sure it is complete as noted in Section A 1-6 (Client's name/ address/ contact name/ phone number/ account information/ PO number/ complete sample information/ analyses requested).

11. All samples are checked to be sure they match the paperwork.

12. The client must be contacted if the information is not complete or if there are any questions about the samples, analyses requested, or if samples are received broken or missing.

**C. Samples received by mail, UPS, Federal Express, etc.**

Samples received by mail, UPS and Federal Express are handled in the same manner as samples received from our drivers with the exception that samples are not relinquished by the client. All coolers received must have a temperature reading as in section B.2. and all samples must be verified against the Chain of Custody or paperwork as noted above.

**D. In-house samples**

In-house samples consist of samples such as QA/QC check samples and hazardous waste disposal samples. These samples are written up using the same procedures as any other sample. (They will not normally be billed.)

**E. Priority samples**

1. Samples are logged in the following priority:

   a. Bacteriology

   b. Rushes (Same Day, 24 Hour, 48 Hour)

   c. Tests such as BOD, Chlorine, pH, Dissolved Oxygen, Sulfite, Sulfide, Hexavalent Chromium, fish toxicity, nitrate, nitrite, MBAS, turbidity must be logged the same day as received due to the very short holding times.

   d. Regular Turn-Around

2. NOTE: It is important that this priority be followed for all customers to insure that accurate results are obtained for samples which have a very short holding time.

3. Regular turn-around samples are written up in the order received and may be held to the next day if necessary.

4. When a client requests a completion date, or we commit to a completion date, this information must be clearly stated (and highlighted) on the lab request summary.

Note: the affected lab manager must be consulted prior to committing to a completion date.

5. If a client wishes samples to be handled on a priority basis, such as 24 or 48 hours, there is an additional charge. The priority charge is determined by lab management, and should be clearly stated to the client.

6. Priority samples are written up and labeled before being transferred to the laboratory. These samples are recorded in the Sample Rush Log Book and the lab personnel receiving the samples must sign for all priority samples (which include a copy of the chain of custody)

**F. Special Handling of Samples for Microbiological Testing**

1. Due to the short holding times for microbiological samples, these must be handled on a first- priority basis

2. The Chain-of-Custody for samples for microbiological testing must state the date and time of sampling, as well as complete sample identification. For potable water samples this should also include the system name and sample location

3. Drinking water samples (potable water) should be analyzed as soon as possible after sampling (30 hours maximum time from sampling to analysis). Samples must be maintained at 4 -10 degrees C during transport and storage. Potable water samples cannot be analyzed after 48 hours, these samples should be refused.

4. Waste water and surface water samples must be analyzed within 6 hours after collection (6 hours maximum holding time). Samples must be maintained at 4 - 10 degrees C during transport and storage. Water/ waste water samples older than six hours should be refused.

5. Upon receipt in Sample Receiving, check samples immediately for proper temperature and holding time. Samples should be transported in a cooler with blue ice or regular ice. Check Chain-of-Custody form to be sure samples are within holding times. If samples are outside holding time or not held at proper temperature, notify the Microbiology Department supervisor or project manager immediately The Chain-of-Custody shall also state the conditions of the samples as received (cooled, frozen, room temp. etc.).

6. Check condition of samples received for microbiological testing for potential contamination of samples. Containers must be sealed with no evidence of leakage. Containers must be protected from melted ice or other potential contamination. Notify the Microbiology supervisor if problems are noted. If there is evidence of contamination the client should be notified that the samples are potentially contaminated.

7. Samples should be refrigerated or placed in a cooler with blue ice upon receipt and logged in immediately. The Microbiology Department will sign the original chain of custody to show receipt of samples prior to logging

**G. Sample storage during login process**

1. When possible samples are written up as soon as received

2. A designated sample storage refrigerator is used for storage of samples which need to be refrigerated during the login process (samples for volatile organics analysis are stored in a separate refrigerator).

3. As soon as possible after each group of samples is logged in, they are transferred to the Sample Custodian in the Sample Storage Area. Most samples are stored in refrigerators or

the walk-in cooler until analyses are completed. The sample storage refrigerators and the walk-in cooler are kept locked overnight for sample security.

H. **Hold samples**

1. When a client wishes to put samples on hold, this must be clearly noted on a Chain-of-Custody form. The length of time requested for hold should be noted.

2. If the hold order is given over the phone, a note is made on the COC referring to the person authorizing the hold, with complete information on the samples to be held. The person taking the call should sign and date the note. Any changes to the Chain of Custody by the client should be followed by a fax from the client detailing the changes in writing.

3. Complete information on hold samples are filed with the Chain-of-Custody and given with the samples to the Sample Custodian for storage until the Client or project manager releases the samples from hold status. If hold samples are disposed of, they are logged out by the Sample Custodian.

4. After 7 days, if the client has not contacted us regarding the samples, sample receiving personnel or the project manager should call the client for instructions.

5. Maximum holding time is 30 days unless special arrangements are made and authorized by the lab management.

6. Unless authorized by the customer, disposal of hold samples must be authorized by the Lab Manager.

I. **Safety Precautions:**

1. The lab does not accept radioactive samples for analysis. A Radiation Monitor is available in the Sample Receiving Department for screening samples if radiation is suspected in any sample

   a. Any samples received from Department of Energy (DOE) contracts or associated clients must be screened to insure that no radioactivity is present.

   b If any sample tests higher than background 25 cpm level radiation, the Radiation Safety Officer must be notified immediately.

2. All sample shipments received from hazardous waste sites or labeled as highly toxic must be initially opened in a fume hood or in a well-ventilated area.

3. Plastic gloves are available in the Sample Receiving Area for handling potentially hazardous samples or samples which are leaking.

4. When in doubt about the safe handling of any sample, the Lab Safety Officer or appropriate Lab Manager must be consulted before the sample is logged in.

## II. CHAIN OF CUSTODY FORM

A. The purpose of the Chain of Custody Form is to legally document the transfer of the sample(s)from the customer to the laboratory. Since any sample may potentially be used as evidence in legal proceedings, it is important that the Chain of Custody Form be filled in completely and accurately.

B. The Chain of Custody Form should furnish an accurate record of the samples received, analyses requested, and any important information from the Client regarding the samples. The information entered on the form should be as complete as possible, including:

1. Client's name and address with zip code

2. Client project manager's name and telephone number

3. Information on custody seals - If present are they intact?

4. *Information on Samples:*

   a. Is the number of samples listed correctly?

   b. Are all samples individual, or sub-samples of one sample?

   c Is the description of the samples complete?
   (are samples soil, waste-water, drinking water (if samples are chemicals, a complete description and MSDS information should be furnished.)

   d. Are samples identified correctly? Sample ID numbers or markings should be checked against the Chain of Custody. The date sampled should also be on the chain of custody.

   e. The condition of the samples should be noted
   - Are samples cool or frozen?
   - Are containers leaking or broken?
   - Damaged containers should be noted on the cooler receipt form under "important information section" and reported to the project manager immediately

   f The type of containers must be noted (glass jar, plastic container, brass tube, VOA vial, etc.)

   g. All preservatives added to the samples must be noted on the sample containers and is indicated on the sample pH log form attached to the chain-of-custody.

   h. Any inconsistencies in the documentation and samples should be thoroughly investigated The ideal time to solve a problem is during the log-in process.

5. Analyses requested by the Client must be specific and correspond EXACTLY to our listed analyses profile. If there is any doubt as to the analyses required, the Sample Receiving

Person should contact the Client, or the appropriate Lab Manager

- In the case where subsamples of the same sample are submitted, and different analyses are requested for each sub-sample, all information and the labeling of each container must be made VERY CLEAR to avoid confusion in the laboratories. EACH CONTAINER MUST HAVE A LAB REQUEST NUMBER and an ORDER NUMBER.

6 Any problems with improper preservation, sample container type, volumes, etc. are to be noted on the Chain of Custody. This is to document problems which may interfere with a proper analysis of the sample. A written copy should also be given to the Lab Project Manager or Customer Representative who may need to contact the customer.

7 The Client should sign in the " Relinquished by " space and also in the " Authorization " space when appropriate.

8 The person receiving the sample(s) must sign the Chain of Custody Form in the "Received by Laboratory for Analysis" space, and record the date and time.

9 When the sample is entered into the Laboratory computer system ( a Lab Request Summary is generated ) the Lab Request Number should be recorded on the Chain of Custody.

10. Distribution of copies:

a. Attach the White and Yellow Copy to the Blue Lab Request Summary

b. The Pink Copy is given to the Client

c. A copy of the Chain of Custody should be attached to all copies of the Lab Request Summary.

d. All Lab Requests are checked by the appropriate Project Manager

III. **SAMPLE CONTROL RECORD** (Internal Chain of Custody)

A. A separate Sample Control Record for sample tracking through the laboratory may be initiated by the Sample Receiving Department if this is required by a client or contract (such as EPA/CLP).

B. Information to be entered into the Sample Control Record (refer to the attached copy):

1. The Lab Request Number is written at the top of the Form

2. The Client's Name and Date is recorded

3. All individual samples are recorded in the Sample ID space. Samples are identified by the Lab Request Number assigned at the time of sample Log-In. This number is generated by the computer when the sample(s) are logged-in to the computer system.

C. Storage of samples requiring Sample Control Record (Legal Samples)

1. After the samples are logged into the computer system and labeled, they are transferred to a locked storage refrigerator in the Sample Storage Area.

2. Document the transfer of all samples to and from the Sample Custodian with the date and time samples were transferred. Both the Sample Receiving person and Sample Custodian sign the Sample Control Record.

3. For Legal Samples (including EPA/CLP samples), the samples must be kept in locked storage. In this case the Sample Control Record is kept by the designated Sample Custodian who also controls access to the samples. When samples are removed from storage they are logged out on the Sample Control Record which records the date, time and person removing the samples. When the samples are returned they are logged back in with the date, time and initials of the person returning the samples. Samples are not removed from locked storage overnight. The person who removes the samples is responsible for the custody of the samples, and for their return to storage before the end of the working day.

D. Sample Control Record Tracking

1. Each time samples are transferred to or from the Sample Custodian, the Sample Control Record for those samples must be signed.

2. Each person receiving the samples in each department must sign for those samples received and also note the date and time samples are received. Fill in Received By - Dept., Person and Date/Time when samples are delivered to each department and again when the samples are returned to the Sample Custodian.

3. Only one sample control record will be completed for each lab request number (Sample Log In Sheet). No copies are to be made unless clearly labeled as a copy.

4. The Sample Control Record is kept on file by the Sample Custodian and attached to the file when all analyses are completed.

## IV. SAMPLE ACCEPTANCE POLICY

Sample acceptance policy determines if the sample is identified correctly, with proper documentation, packaging, adequate volume for the analyses requested and correct preservatives.

1. Sample identification ( is the sample waste water, drinking water, hazardous waste, unknown? ). For accurate analysis, the sample and sample source must be identified correctly. If there is an obvious discrepancy between the sample and documentation, this is normally investigated first by the Sample Receiving Personnel. If the problem cannot be resolved, then the appropriate lab manager is notified.

2. Documentation with the sample ( is it adequate? ). Sufficient documentation should be supplied with the sample to fill in the Chain of Custody completely. If there are any doubts as to the sample identification or analyses requested, the client should be called immediately.

3. Documentation generated during sample login. All communications and decisions regarding the client samples should be documented and signed in writing and attached to the original Lab Sheet (and all copies if necessary)

4. Sample condition (sufficient volume, correct preservative, correct container type, condition of sample, etc). The employee receiving the sample must note on the Chain-of-Custody form or an attached Sample Receiving form the following information for each sample and fraction:

   a. Container Type (Glass, Amber glass, plastic, brass tube, etc )

   b. Volume in container (1 L, 500 ml, etc.)

   c. Temperature (Room temp., cool, frozen)

   d. If samples are in a cooler, the temperature in the cooler

   e. Preservatives added must be listed on the sample container and/or the Chain of Custody form.

   f. The sample must be within the specified holding times for the analyses requested.

   g. Any irregularities noted in the samples (leaking, air bubble in VOA vial, improper packaging, etc.).

5. Responsibility for contacting the customer about problems. The Sample Receiving personnel have primary responsibility for contacting the project manager or client immediately for routine problems with samples. Each client is normally assigned to a project manager, and the person logging the sample is also responsible for informing the project manager of any problems. This may be done with notes on a copy of the lab sheet or chain of custody. Generally all information and decisions must be documented in writing with a date and signature.

## V. SAMPLE LOGGING PROCEDURES

### A. Description of Computer Logging Procedure:

1. The LIMS system will be used to record and track all samples received at the laboratory. Completed test results should be turned in to the project manager as designated on the Lab Request Summary

2. Each Department should report the results of all analyses on the blue Results Worksheet and turn this in to the project manager, along with all worksheets and raw data generated in analyzing the samples.

3. When samples are logged into the LIMS system, the system will create one label for each sample container, a Lab Request Summary on blue paper, and a Results Worksheet for each lab department on blue paper. When samples are logged into the LIMS, they are assigned a unique sample number (order number) and all samples in the same group, received on the same day are normally assigned to a unique Lab Request Number.

4. The Sample Receiving personnel will make copies of the login documents as follows: A copy of the Lab Request Summary and the chain-of-custody for each Results Worksheet.

5. Copies of the login documents will be distributed as follows:

   a. Project Manager: The Lab Request Summary and one copy of the Chain of Custody.

   b. Each Department: The blue (original) Results Worksheet + copy of the Lab Request Summary + copy of the Chain of Custody.

   c. Attach the original Chain of Custody to the original Lab Request Summary.

   d. A Posting Log Book is maintained to verify that a copy of the Lab Request and Worksheets was distributed to each affected Department.

6. If problems are noticed with the test codes, analyte list or detection limits ( DLR ) please correct the Worksheet and give a copy to Jim or Steve as soon as possible so corrections can be made in the LIMS.

B. **Description of Lab Request Summary**

1. A Lab Request Summary is prepared which includes:

   a. Client name, address and client ID number.

   b. Person to whom final report is to be sent.

   c. Date sample received.

   d. A complete description of the sample(s) including client identification number(s), sample matrix, date /time sampled.

   e. A Lab Request Number and an order Number is generated by the computer for each sample.

f. A complete list of all analyses to be completed on each sample, including Method Number, Profile and Service Group / Department.

g. Login information including ID of person logging in the sample, date, time and if automatic analysis selection was used.

h. Order numbers and corresponding customer ID numbers for each sample.

i. A Sample Control Record (Internal Chain of Custody) is completed if needed. This document is used to record the transfer of the samples to departments (see section III).

**C. Sample Labeling**

Each sample is labeled with the label generated by the computer. The label contains the Lab Request Number, Order Number, Client sample ID and log date.

*For Orders where multiple containers are submitted (multiple fractions for different analyses), each separate container (fraction) should be labeled with the order number + A, B, C, etc. to designate fractions for each separate analysis. This fraction designation is then recorded by the custodian and analyst on the sample preparation log to document that the correct sample fraction was analyzed for each analysis method.*

**D. Procedure for Logging in Additional Analyses.**

1. If additional analyses are requested by a client after the samples have been initially logged in and distributed to the labs, an amended Lab Request Summary may be generated for the additional analyses (using the same Lab Request number). The amended Lab Request Summary will note the additional tests in the Comments section.

2. Additional analyses may also be noted using a yellow edit / additional analyses request form to notify all affected departments of the additional tests. Information required is as follows:

a. Name of client
b. Previous Lab ID#
c. Sample type
d. Sample ID
e. Additional analyses
f. Date of request
g. Signature of employee

3. A new Lab Request will be generated if necessary. The new Lab Request Summary will have a new Lab Request Number for the additional analyses, and the samples will be relabeled with the new Lab Request Number. The original Lab Request Number will be retained on the samples.

a. The new Lab Request Summary must clearly reference the original Lab Request number and explain that analyses requested are in addition to the previous analyses (or other reasons for the new Lab Request Summary).

b. Copies of the new Lab Requests are forwarded to all departments affected.

E. **Backup Logging Procedure in Event of Computer System Failure.**

1. Temporary lab Request Summaries have been designed and are available in the Sample Receiving Department.

2. In the event the computer system is non-functional, the Sample Receiving Supervisor will issue temporary lab Request Summaries along with a temporary login reference number (eg. A100).

3. The supervisor will keep a list of assigned numbers and corresponding information (client, departments receiving lab Request Summaries, person writing the ticket).

4. When the computer is functional, standard lab Request Summaries will be issued.Samples that have received temporary numbers will be retrieved and re-numbered with the computer assigned lab Request Numbers. The standard lab Request Summaries will be attached to each corresponding temporary lab Request Summary that was issued.

## VI. HANDLING OF THE SAMPLES AFTER LOGGING

### A. Handling of the logged-in samples in the laboratory

1. After the samples are logged into the computer system and labeled, they are transferred to the Sample Custodian in the Sample Storage Area. All samples are logged into the Sample Control Log Book organized by Lab Request number. The client name, number and type of containers are entered. The Sample Custodian must sign the Log Book for all containers received.

2. The samples are stored in locked refrirators or the locked walk-in cooler prior to analysis.

3. All samples transferred to the Sample Storage Area are logged into a Sample Logbook in the Sample Storage Area. The Sample Logbook is maintained by the Sample Custodian.

4. When samples are picked up by laboratory personnel for analyses, the samples are signed out, and when returned, they are signed back into Sample Storage.

5. When samples are disposed of, this is noted in the Sample Logbook.

6. During weekends and evenings, only designated personnel have access to the

Sample storage areas. All samples removed must be documented in the Sample Custodian Logbook.

**B. Handling of samples to be sent out to other labs.**

1. Arrangements to send samples out for analysis are handled by the project manager and must have the Client's consent.

2. Samples to be transferred to another lab are logged into the LIMS for "Send Out" and the Information is posted on the "Out Board" similar to posting to an in-house department. Samples to be sent out are subsampled and shipped by the Sample Custodian.

3. A portion of each sample to be sent out is retained in the original container.

**C. Returning samples to the client.**

1. When a client requests that the samples be returned to them upon completion of the analyses, the sample receiving personnel should make sure that a notification is made on the lab sheet and that it is clearly visible

2. When all analyses are completed, a note is given to the Sample Custodian listing the samples to be returned and address to be used.

3. If the sample is returned by UPS, the sample pickup record will document that the sample was returned. If the sample is delivered by our driver or picked up by the client, the client should sign the chain of custody or a receipt to show the samples were returned to them. A record book is maintained in Sample Receiving to document the return of samples.

**APPENDIX C**

# Sample Container and Preservation Guide

***Updated: March 20, 2002***

| | Method | Container(1) | Suggested Volume | Preservative | Holding Time(2) |
|---|---|---|---|---|---|
| **Volatile Organics** | | | | | |
| (VFH) Gasoline | (5030) 8015 Mod | VOA-glass | 2 40ml vials | Cool 4 C | 7 days(3)/14 soil |
| (VFH) Gasoline/BTEX | (5030) 8015/8020 | VOA-glass | 2 40ml vials | Cool 4 C | 7 days(3)/14 soil |
| Halocarbons | 601/8010 | VOA-glass | 2 40ml vials | Cool 4 C | 14 days |
| Aromatics | 602/8020 | VOA-glass | 2 40ml vials | Cool 4 C | 7 days(3)/14 soil |
| Purgeables | 624/8240 | VOA-glass | 2 40ml vials | Cool 4 C | 14 days |
| Purgeables in DW | 524.2 | VOA-glass | 2 40ml vials | Cool 4 C | 14 days |
| **Semi-Volatile Organics** | | | | | |
| (EFH) Diesel | 8015 Mod | glass-amber | 1 L | Cool 4 C | 7 days/14 soil(4) |
| Semi-Volatiles (BNAs) | 625/8270 | glass-amber | 1 L | Cool 4 C | 7 days/14 soil(4) |
| Pesticides & PCBs | 608/8080 | glass-amber | 1 L | Cool 4 C | 7 days/14 soil(4) |
| Phosphorous Pests. | 614, 622/8140 | glass-amber | 1 L | Cool 4 C | 7 days/14 soil(4) |
| Herbicides | 615/8150 | glass-amber | 1 L | Cool 4 C | 7 days/14 soil(4) |
| Polynuclear Aromatics | 610/8100 (8310) | glass-amber | 1 L | Cool 4 C | 7 days/14 soil(4) |
| Haloacetic Acids | 552.2 | glass-amber | 250 ml | Cool 4 C, 5mg NH4Cl/50ml | 14 days(4) |
| Carbamate Pesticides | 632 | glass-amber | 1 L | Cool 4 C | 7 days(4) |
| **Metals** | | | | | |
| Mercury | 245.1/7471 | poly | 500 ml | HNO3 to pH<2 | 28 days |
| Chromium VI | 218.4/7196 | poly | 500 ml | Cool 4 C | 24 hours |
| Organic Lead | DHS (LUFT) | glass-amber | 1 L | Cool 4 C | 14 days |
| All Other Metals | 200/6000/7000 | poly | 500 ml | HNO3 to pH<2 | 6 months |
| **Inorganic & Wet Chemistry** | | | | | |
| Alkalinity | 310.1 | poly or glass | 500 ml | Cool 4 C | 14 days |

| | | | | | |
|---|---|---|---|---|---|
| COD | 410.4 | poly or glass | 500 ml | Cool 4 C, H2SO4 to pH<2 | 28 days |
| Chloride | 300 | poly or glass | 500 ml | None | 28 days |
| Cyanide | 335.1/335.2/9010 | poly or glass | 1 L | Cool 4 C, NaOH to pH>12(5) | 14 days |
| Flashpoint | 1010 | poly or glass | 500 ml | None | N/A |
| Fluoride | 300.0/340.2 | poly or glass | 500 ml | None | 28 days |
| Hardness | 130.2/SM314B | poly or glass | 500 ml | HNO3 or H2SO4 to pH<2 | 6 months |
| Nitrate/Nitrite | 353.2 | poly or glass | 500 ml | Cool 4 C | 48 hours |
| Oil & Grease | 413.1/413.2 | glass-amber | 1 L | Cool 4 C, H2SO4 to pH<2 | 28 days |
| Phenols | 420.1 | glass-amber | 1 L | Cool 4 C, H2SO4 to pH<2 | 28 days |
| Phosphorous | 365.2 | poly or glass | 500 ml | Cool 4 C, H2SO4 to pH<2 | 28 days |
| PH | 150.1 | poly or glass | 500 ml | None | Immediate |
| Solids (IDS, TSS, IS) | 160.1/160.2/160.3 | poly or glass | 500 ml | Cool 4 C | 7 days |
| Specific Conductance | 120.1 | poly or glass | 500 ml | Cool 4 C | 28 days |
| Specific Gravity | SM2710F | poly or glass | 500 ml | None | N/A |
| Sulfate | 375.4 | poly or glass | 500 ml | Cool 4 C | 28 days |
| Sulfide | 376.2 | poly or glass | 500 ml | Cool 4 C, ZnCO2CH3+NaOH pH>9 | 7 days |
| IRPH | 418.1 | glass-amber | 1 L | Cool 4 C, H2SO4 to pH<2 | 28 days |
| TOC | 415.1 | glass-amber | 250 ml | HCL to pH<2 | 7 days |
| TOX | 9020 | glass-amber | 500 ml | HNO3 to pH<2 | 28 days |
| Radioactivity | 900 | Any | 1 L | HNO3 to pH<2 | 7 days |
| Bioassay (Effluent) | 600/4-85/01 | poly or glass | 5 Gallons | Cool 4C | 24 hours |

**Notes:**

(1) Soil samples are typically collected in brass or steel tubes and wide mouth jars (500ml)

(2) Unless otherwise stated, holding times apply to soil and water matrices.

(3) To extend the holding time to 14 days, prepare bottle with HCL to pH<2

(4) Holding times shown are days until extraction. Samples have a 40-day (7-day for 552.2) holding time after extraction.

(5) If chlorinated, add 0.6g Ascorbic Acid

**APPENDIX D**

# Capital Equipment Inventory

***Last Update: October 14, 2004***

| Department | Instrument Description | Quantity | Serial No. | Date |
|---|---|---|---|---|
| Chemistry | Perkin Elmer FIMS400 Flow Injection Mercury Analyzer with AS90 Autosampler and Data System | 1 | 4543 | |
| Chemistry | Lachat FIA+ Quickchem 8000 Flow Injection Analyzer with Autosampler and Data System | 1 | A83000-1315 | |
| Chemistry | Lachat Colorimeter (10mm path) | 1 | | |
| Chemistry | Lachat Manifold (NO2/NO3) | 1 | 10_107_04_O | |
| Chemistry | Lachat Manifold (NH3-N) | 1 | 10_107_06_1-A | |
| Chemistry | Lachat Manifold (TKN) | 1 | 10_107_06_2-E | |
| Chemistry | Lachat Manifold (CN) | 1 | 10_204_00_1-A | |
| Chemistry | Lachat Manifold (TKP) | 1 | 10_115_01_1-P | |
| Chemistry | Dionex 2000 Ion Chromatograph with Autosampler, ASRS Supressor, CD20 Conductivity Detector and data system – System I | 1 | 96030596 | |
| Chemistry | Dionex 2000 Ion Chromatograph with Autosampler, ASRS Supressor, ED40 Electrochemical Detector and data system – System II | 1 | 97020907D991001 | |
| Chemistry | Dionex 2000 Ion Chromatograph with Autosampler, ASRS Supressor, CD25 Conductivity Detector and data system (perchlorate analysis) – System III | 1 | 01090605 | |
| Chemistry | Dionex 2000 Ion Chromatograph with Autosampler, AD25 Absorbance Detector and data system (hexavalent chromium analysis) – System IV | 1 | 01120109 | |
| Chemistry | Tekmar Dohrman DX-2000 TOX Analyzer with data system | 1 | 98023001 | |
| Chemistry | Beckman DU-50 Spectrophotometer | 1 | 4640062 | |
| Chemistry | Thermo Electron Spectrophotometer | 1 | | 2004 |
| Chemistry | Mettler AE163 Scale | 1 | D14314 | |
| Chemistry | Mettler AE163 Scale | 1 | WB1225 | |

| | | | | |
|---|---|---|---|---|
| Chemistry | Mettler AE200 Scale | 1 | J79480 | |
| Chemistry | Mettler PE3000 Scale | 1 | F17120 | |
| Chemistry | Sartorius B310P Scale | 1 | 38060060 | |
| Chemistry | Sartorius BA61 Scale | 1 | 30701480 | |
| Chemistry | Labconco 65200-00 Rapidstill II | 1 | 990192069E | |
| Chemistry | Fisher Scientific Coulomatic K-F Titrimeter | 1 | 842 | |
| Chemistry | Beckman TJ-6 Centrifuge | 1 | 7A055 | |
| Chemistry | Eppendorf 5415C Centrifuge | 1 | 5415B67934 | |
| Chemistry | Fume Hoods | 6 | | |
| Fish Toxicity | 4 Gallon Tanks | 40 | | |
| Fish Toxicity | Disposable Tanks (approx. 3 Gallons each) | 100 | | |
| Fish Toxicity | 30 Gallon Tank | 3 | | |
| Fish Toxicity | 20 Gallon Tank | 1 | | |
| Fish Toxicity | Air Pumps | 10 | | |
| Fish Toxicity | Circulation Pump | 1 | | |
| Fish Toxicity | pH Meter | 1 | | |
| Fish Toxicity | Recording Thermograph | 1 | | |
| Fish Toxicity | YSL Model 50B DO Meter | 1 | | |
| GC | Varian 3400 GC with FID & PID (#3w) | 1 | | 1991 |
| GC | Varian 3400 GC with FID & PID, autosampler and data system (# 1) | 1 | | 1989 |
| GC | Varian 3300 GC with FID & PID, autosampler and data system (# 2) | 1 | | 1989 |
| GC | Varian 3400 GC with FID, autosampler and data system (# 3) | 1 | | 1989 |
| GC | Varian 3400 GC with FID, autosampler and data system (# 9) | 2 | | 1986 |
| GC | Varian CP-3800 GC with FID & PID, autosampler and data system (# 14) | 1 | | 1999 |
| GC | Varian CP-3800 GC with FID & PID, autosampler and data system (# 15) | 1 | | 2004 |
| GC | Varian 3300 GC with FID, autosampler and data system (# 12) | 1 | | 1986 |
| GC | Varian 3400 GC with PID (Traacor 1000 HALL Detector attached), autosampler and data system (# 4) | 1 | | 1989 |
| GC | Varian 3400 GC with PID & FID, autosampler and data system (# 5) | 1 | | 1990 |
| GC | Varian 3400 GC with ICD (# 8) | 1 | | 1988 |
| GC/MS | Varian Model 3800 gas chromatograph with Varian Saturn 2200 MS Detector, Autosampler and Data Station (#7) | 1 | 04575-10060 | 2003 |
| GC/MS | Varian Model 3800 gas chromatograph with Varian Saturn 2000 MS Detector, Autosampler and Data Station (#6) | 1 | 4443-6028 | 2001 |

| | | | | |
|---|---|---|---|---|
| GC/MS | Varian Model 3800 gas chromatograph with Varian Saturn 2000 MS Detector, Autosampler and Data Station (#5) | 1 | 3810-3780 | 1999 |
| GC/MS | Varian Model 3800 gas chromatograph with Varian Saturn 2000 MS Detector, Autosampler and Data Station (#4) | 1 | 3811-3781 | 1999 |
| GC/MS | Varian Model 3400 gas chromatograph equipped with Varian Saturn Model 2000 MS Detector (#3) | 1 | | |
| GC/MS | Hewlett Packard 5890 gas chromatograph with a Hewlett Packard 5971 Mass Selective Detector and a HP 7673A automatic injector | 1 | | |
| GC/MS | Varian Model 3800 gas chromatograph equipped with Varian Saturn Model 2000 MS Detector, 2 flame ionization detectors, and a Lotus air sampling system | 1 | | |
| Microbiology | Castle Thermatic 60, 20x24 Autoclave, Automatic | 1 | | |
| Microbiology | Market Forge Sterilmatic Autoclave | 1 | | |
| Microbiology | Wesco, 4 Objective Microscope | 1 | | |
| Microbiology | B&L Dissecting Microscope | 1 | | |
| Microbiology | "Filamatic" Media Pipettor | 1 | | |
| Microbiology | Blue M Magni-Whirl Constant Temperature Bath | 3 | | |
| Microbiology | Neslab 500 Water Bath | 3 | | |
| Microbiology | Lab-Line Imperial III Incubator | 1 | | |
| Microbiology | Thelco Incubator | 1 | | |
| Microbiology | Precision Scientific Incubator | 1 | | |
| Microbiology | Baush & Lomb Refractometer | 1 | | |
| Microbiology | VWR 1555 Incubator | 1 | | |
| Microbiology | VWR Incubator, 40 cubic ft. | 1 | | |
| Microbiology | Lab-Line Orbiter Environmental Shaker | 1 | | |
| Microbiology | Bio-Rad Mini-Transilluminator | 1 | | |
| Microbiology | Bio-Rad AC Power Supply | 1 | | |
| Microbiology | Baxter Scientific Product Vortex Mixer | 1 | | |
| Microbiology | Sartorium Universal Balance | 1 | | |
| Microbiology | Quebec Colony Counter | 1 | | |
| Office Data Handling | Toshiba Fax | 2 | | |
| Office Data Handling | Canon Copiers | 4 | | |
| Office Data Handling | LIMs Computer System (39 stations) | 1 | | |
| Office Data Handling | HP Laserjet Printers | 3 | | |

| | | | | |
|---|---|---|---|---|
| Office Data Handling | Lexmark Printers | 13 | | |
| Pesticides | Hewlett Packard 5890A Series II GC, dual ECD detectors, Autosampler and Data Station | 1 | 3022A28956 | 1990 |
| Pesticides | Varian 3400 GC, dual ECD detectors, Autosampler (GC-3400) | 1 | 14304 | 1991 |
| Pesticides | Varian 3800 GC, dual ECD detectors, Autosampler (GC#1) | 1 | 2771 | |
| Pesticides | Varian 3800 GC, dual ECD & PFPD detectors, Autosampler (GC#2) | 1 | 6056 | 2000 |
| Pesticides | Varian 3800 GC, dual ECD & PFPD detectors, Autosampler (GC#3) | 1 | 9085 | 2000 |
| Pesticides | Varian 3400 GC, FID detector, Autosampler (GC-Alcohol) | 1 | 6692 | 1989 |
| Pesticides | Waters Dimension II GC, ECD & FID detectors, data system | 1 | GC2-8901009 | |
| Pesticides | Varian 9100 HPLC with UV-Vis, RI, Fluorescence Detectors, Autosampler and data system | 1 | 3021 | 1994 |
| Pesticides | Shimadzu SCL-10A VP System Controller, LC-10A1 Pumps, Autosampler, SPD-M10A VP Diode Array Detector, Data System | 1 | C2103750927US | 2000 |
| Pesticides | Shimadzu GC-2010, dual injectors, dual ECD detectors (ECD#1, ECD#2), Autosampler and workstation | 1 | C11324101922 | 2003 |
| Pesticides | Dionex ASE 200 Accelerated Solvent Extractor and Controller | 1 | 1060057 | 2001 |
| Pesticides | Dionex ASE 200 Accelerated Solvent Extractor and Controller | 1 | 97060620 | 2000 |
| Pesticides | Zymark Turbo Vap II Concentration Workstations | 3 | | 2000 |
| Pesticides | Ohaus Brainweight B1500D Toploader Balance | 1 | 11532 | |
| Pesticides | Boekel 1494 Steambath | 1 | | |
| Pesticides | Fisher Isotemp 228 Steambath | 2 | | 2000 |
| Pesticides | Fume Hoods | 5 | | |
| Pesticides | Varian 3300 GC (Drying Oven) | 1 | 5415 | 1988 |
| Pesticides | B. Braun Braun-Sonic U Ultrasonic probe and generator | 1 | | |
| Pesticides | VWR 1350G Drying Oven, gravity | 1 | | |
| Pesticides | Precision Scientific 16 Drying Oven, gravity | 1 | | |
| Pesticides | National Appliance Drying Oven, gravity | 1 | | |
| IOC/RAD | Gas-Flow proportional counting system -- Protean Instr., Model 9025. | 1 | | 1991 |
| IOC/RAD | Geiger-Mueller Counter (portable) -- S.E. Intl. Model 4EC | 1 | | 1991 |

| | | | | |
|---|---|---|---|---|
| IOC/RAD | Infrared Heater and Stand (Fisher Scientific, Model 11-504-5 | 1 | | 1991 |
| IOC/RAD | Labconco Model 59000 Chemical Fume Hood | 1 | | 1991 |
| IOC/RAD | Mettler Model H35AR Analytical Balance | 1 | | |
| TOC/RAD | Dessicator, Nalgene Model 8-642-21 | 1 | | 1991 |
| TOC/RAD | TOC Analyzer, Shimadzu, TOC-5000 | 1 | | |
| IOC/RAD | Shimadzu TOC-VCSH Total Organic Carbon Analyzer, A/S and Data System | 1 | | 2004 |
| AA/ICP Metals | PE Sciex Elan 6100 ICP-MS with auxiliary data system and autosampler | 1 | | 2000 |
| AA/ICP Metals | Perkin Elmer Optima 4300DV ICP with autosampler and data system | | | 2001 |
| AA/ICP Metals | Perkin Elmer Aanalyst 100 AA | 1 | | 2001 |
| AA/ICP Metals | TCLP Rotary Agitators | 2 | | |
| AA/ICP Metals | TCLP ZHE Extractors | 4 | | |
| AA/ICP Metals | TCLP Pressure Filters | 2 | | |
| AA/ICP Metals | Fume Hoods | 2 | | |
| AA/ICP Metals | Environmental Express Hot Blocks | 3 | | |
| | | | | |

## APPENDIX E

## STANDARD OPERATING PROCEDURE FOR DETERMINATION AND UPDATING OF MDL/DLR DETECTION LIMITS

### PURPOSE

1. This Standard Operating Procedure summarizes the procedure for determining MDLs (Method Detection Limit) and DLR (Reporting Detection Limit), in addition to the procedure for updating and revising current MDLs and DLRs.

### DETERMINATION OF MDL

1. Prepare and analyze seven replicate spike solutions:

   1.1. Prepare one spiked bulk solution for each matrix at 1-5 times the estimated detection limit. The volume should be sufficient to prepare and analyze seven or more samples. The solution should be spiked with all analytes of interest.

   1.2. Prepare seven or more aliquots of the spiked solution per the normal method of preparation (process through the entire analytical method).

   1.3. Analyze all the aliquots by normal analysis procedures (QA samples such as spikes, duplicates, LCS and PB are not required).

   1.4. Calculate the standard deviation (n-1) of the seven results. For seven replicates multiply by 3.14 to calculate the MDL value for each analyte. (**NOTE:** Use the factor 3.14 only for seven replicates, other factors are given in the EPA reference noted below).

   1.5. More than 7 aliquots can be analyzed. If more than 7 aliquots are analyzed, then all values must be used in calculating the MDL. Use the Student's t value at the 99% confidence level for the number of replicates.

2. The MDL should be determined at least once a year for each analyte and each analytical method. The MDL should be re-run whenever there is a significant change in instrumentation or procedure.

3. An MDL check sample at approximately 2 x MDL should be analyzed to verify the reasonableness of the MDL values obtained. The MDL check sample should be prepared the same way as the MDL check solutions. All analytes should be detected in the MDL check sample, or the MDL study should be modified and repeated for the analytes which are not detected.

## DETERMINATION OF REPORTING DETECTION LIMIT (DLR)

1. Prepare and analyze one or more samples at the estimated reporting limit:

   1.1. Prepare one or more samples at the estimated reporting limit using the normal preparation procedure (process through the entire analytical method). QA samples such as spikes, duplicates, LCS and PB are not required.

   1.2. Analyze the sample by the normal analysis procedure.

   1.3. The analytical result must be 75-125 percent of the spike value. If not, increase the concentration until this accuracy can be achieved.

2. The concentration at which the spike recovery of 75-125% can be achieved is the Reporting Detection Limit (DLR).

## UPDATING & REVISING MDL/DLR VALUES:

1. Every year, each department is required to submit their MDLs for each analyte and each analytical method to the QC department.

2. The QC department will then incorporate the current MDLs into the LIMS system for each analytical method (**NOTE:** In the LIMS, there may be several test codes for a particular analytical method. It is important that the MDLs for ALL test codes in the LIMS be updated).

3. After the MDLs for a particular test have been changed, the specs for that test are printed out and kept on file by the QC department, and a copy is returned to the analyst.

4. The QC department shall keep track of all changes in the MDLs through an MDL Master Tracking List, which contains the following information:

   4.1. The date the MDL for a particular test was updated.

   4.2. The date the MDL was run.

   4.3. The LIMS test code and test name for each test in which the MDLs have been updated.

   4.4. The corresponding analytical method for each test.

   4.5. Any additional comments for documenting any pertinent information or noting any unusual peculiarities in the database (e.g., some analytes that are missing DLRs, MDLs that are greater than the DLR, etc.).

5. The MDL should always be less than the DLR. In some cases, the MDL may be equal to or greater than the DLR. In such cases, the following steps must be taken:

   5.1. If the MDL is greater than the DLR for one or more analytes, then the MDL should be

re-run or the DLR should be adjusted if possible.

5.2. If the MDL is equal to the DLR, then this must be reviewed by the QC department as well as the department supervisor to determine if such a scenario is acceptable.

5.3. All cases in which the MDL is greater than or equal to the DLR, including any steps taken to remedy the situation, must be noted in the MDL Master Tracking List.

**REFERENCES:**

1. 40 CFR, Chapter 1, Pt. 136, App.B (7-1-86 Ed).

2. NELAC Quality Systems Revision 16, July 12, 2002.

## APPENDIX F

## NON-CONFORMANCE CRITERIA AND DOCUMENTATION PROCEDURES

### QA Samples - Corrective Actions:

1. Lab Control Sample (LCS- W for water samples, S for soil samples), the acceptance criteria for the LCS is 80 - 120 percent of true value or the current control limits. If not, all samples in the batch must be re-prepared and re-analyzed.

2. Method Blank (MBW for water samples, MBS for soil samples), the result must be less than the reporting limit for each element, or less than 1/10 the lowest sample in the batch. If not, all samples in the batch must be re-prepared and re-analyzed.

3. Matrix Spike Sample (MS), recovery should be 75 - 125, if not the sample result should be flagged for potential matrix interference for each element showing poor recovery. (For metals analyses, a post- digestion spike should be done for any element with poor matrix spike recovery).

4. Matrix Spike Duplicate (MSD), the relative percent difference between the MS and MSD should be less than 20 percent. If not the analysis should be repeated or the result flagged for precision out of limits.

5. Surrogate Recovery, the surrogate recoveries should be within the current control limits for all methods where surrogate recoveries apply. If the surrogate recoveries are outside control limits, the results should be flagged for potential matrix interference for each analyte showing recovery outside the control limits. If the surrogate recoveries for the LCS or Method Blank are outside control limits, all samples in the batch must be re-prepared / re-analyzed, unless it can be determined that the poor recovery was due to a problem specific to that sample only.

### Non-conformance Documentation Form (NCD):

1 Non-conformances such as QA limit failures which can not be corrected by re-analyses, client requirements which cannot be met or standard method modifications are documentated by initiating a Non-Conformance Document Form (NCD). A copy of the NCD Form is attached.

2 The NCD form is initiated by the analyst in the event of a QC sample exceeding control limits or other known non-conformance to the analytical method or client requirements. The NCM may also be initiated by the project manager or department manager in the event client requirements are not met or other analytical problems are discovered.

3 After the NCD Form is initiated, the corrective action must be determined and agreed

upon by the department manager or supervisor and the QA Manager. This is documented and signed by the department manager in the second part of the NCD Form. The form is then forwarded to the QA Manager.

4. The QA Manager then completes and signs the final part of the form. If necessary, verification of the corrective action is documented in this section.

5. A copy of the form is included in the affected data package or the client is notified as appropriate. The original is filed in the Corrective Actions File which is maintained by the QA Manager.

# Associated Laboratories

Non-Conformance Document

Date: _____
Lab Request: _____
Client ID: _____
Department: _____

Document File #: _____
Type of NCD: _____
*(QA Limits, Client Req, Other)*

| Description of Non-Conformance: |
|---|
| |
| |
| |
| |
| Signed (Initiator) _____ Date: _____ |

| Description of Corrective Action: |
|---|
| |
| |
| |
| |
| |
| Signed (Supervisor): _____ Date: _____ |

| QA Manager Approval: |
|---|
| |
| |
| |
| Signed (QA Manager): _____ Date: _____ |